# ChemComm

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2461

2465

<sup>1</sup>Cr(CO)<sub>3</sub>

HO

Ċr(CO)<sub>3</sub> OH

#### Cover



Chiral arene chromium ligands against a backdrop of the London Eye, which represents the continuous cycling of these ligands in a catalytic reaction. Photograph courtesy of Shay Goldschmidt from Herzliya in Israel (http://community.webshots.com/user/shaygold).

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# <u>iontents</u>

## Molecules that add up

Gareth J. Brown, A. Prasanna de Silva and Sara Pagliari



. Ċr(CO)₃

Cr(CO)<sub>3</sub>

Development of molecular systems using chemical imputs and light output, suited to small-scale computational processing.

#### EATURE ARTICLE

#### Asymmetric catalysis using planar chiral arene chromium complexes

Susan E. Gibson and Hasim Ibrahim

Recent developments in the chemistry of planar chiral arene chromium complexes demonstrate that these complexes are emerging as a valuable and versatile ligand class for asymmetric catalysis.



OPCp<sub>2</sub>

PCp<sub>2</sub>

#### COMMUNICATIONS

Synthesis and luminescence properties of mesostructured thin films activated by *in-situ* formed trivalent rare earth ion complexes

Michael H. Bartl, Brian J. Scott, Howard C. Huang, Gernot Wirnsberger, Alois Popitsch, Bradley F. Chmelka and Galen D. Stucky\*

Complexes with trivalent rare earth ions and 1,10-phenanthroline were formed *in-situ* during syntheses of mesostructured silica thin films. This simple one-step synthesis approach results in stongly luminescent materials with high spectral purities of the emitted light.

i



OMON

## The first direct probing of porosity on supported mesoporous silica thin films through hyperpolarised <sup>129</sup>Xe NMR

Andrei Nossov, Elias Haddad, Flavien Guenneau, Claude Mignon, Antoine Gédéon,\* David Grosso, Florence Babonneau, Christian Bonhomme and Clément Sanchez\*

Hyperpolarized <sup>129</sup>Xe NMR spectrum of xenon adsorbed on silica mesoporous films and corresponding transmission electron microscopy (TEM) image.

#### Total synthesis of coleophomone D

K. C. Nicolaou,\* Tamsyn Montagnon and Georgios Vassilikogiannakis

A concise total synthesis of coleophomone D, a natural product that exists as a dynamic mixture of four isomeric compounds, by a strategy based on an acyl cyanide coupling reaction to assemble the key tricarbonyl motif, is reported.

# 2480

## An expedient entry into the fused polycyclic skeleton of vannusal A

K. C. Nicolaou,\* Michael P. Jennings and Philippe Dagneau

The synthesis of the fused polycyclic carbon framework of vannusal A is described. Key features of the synthetic strategy include inter- and intramolecular aldol reactions, a Mn(III) initiated radical cyclization, and a ring-closing olefin metathesis reaction.

## Formation of novel diaza-metallacycles by insertion of tungsten(II) aryloxides into aromatic diazine rings

Margaret R. Lentz, Phillip E. Fanwick and Ian P. Rothwell\*



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A novel series of metallacycles have been generated *via* insertion of a W(II) aryloxide into the N=N bond of aromatic diazines.

## Molecular squares, rectangles and infinite helical chains utilising the simple 'corner' ligand 4-(2-pyridyl)-pyrimidine

Derek A. Beauchamp and Stephen J. Loeb\*

The ligand 4-(2-pyridyl)-pyrimidine forms multinuclear Ag(I) complexes by a combination of chelating and bridging coordination modes; molecular shape (square or rectangle) and degree of aggregation depend on the anion used.

ii



#### A molecular sphere of octahedral symmetry

Dillip Kumar Chand, Kumar Biradha, Makoto Fujita,\* Shigeru Sakamoto and Kentaro Yamaguchi

A tridentate ligand and Pd(II) ion are quantitatively self-assembled into a molecular sphere, which has been characterized by NMR, CSI-MS, and X-ray diffraction study.



OR

Cu2-

minin

Cu

Na

Cu Na Na<sup>+</sup> 2490

2492

ÒR

4Å MS

R: <sup>t</sup>BuMe<sub>2</sub>Si > Et<sub>3</sub>Si > <sup>i</sup>Pr<sub>3</sub>Si > Me<sub>3</sub>Si

-lvdrazine

Nat

## Nickel-loaded $La_2Ti_2O_7$ as a bifunctional photocatalyst

Jindo Kim, Dong Won Hwang, Hyun-Gyu Kim, Sang Won Bae, Sang Min Ji and Jae Sung Lee\*

Nickel-loaded  $La_2Ti_2O_7$ , one of the highly donor-doped (110) layered perovskite materials, could be utilized as the bifunctional photocatalyst for simultaneous  $H_2$  production from water splitting and decomposition of TMAH under UV irradiation.

## Extension of ring closing metathesis methodology to the synthesis of carbocyclic methyl and silyl enol ethers

Varinder K. Aggarwal\* and Adrian M. Daly

Carbocyclic methyl and silyl enol ethers have been successfully synthesised using the second generation Grubbs ring closing metathesis catalyst. When the substrate does not bear any *gem* disubstitution along the alkyl chain, molecular sieves and the size of the silyl group, play a vital role in the success of the reaction.

## One-dimensional organization of copper nanoparticles by chemical reduction of lipid-copper hybrid nanofibers

Masaki Kogiso,\* Kaname Yoshida, Kiyoshi Yase and Toshimi Shimizu

One-dimensional organization of copper nanoparticles has been achieved by chemical reduction using lipid-copper hybrid nanofibers as a template; the reduction of copper ions and the resulting formation of copper clusters occurred at intervals of 2–5 nm along the nanofibers.



#### Solid phosphorus carbide?

Frederik Claeyssens, Neil L. Allan,\* Paul W. May, Pablo Ordejón and Josep M. Oliva

Density-functional theory indicates  $P_4C_3$  will adopt structures quite different from carbon nitride  $C_3N_4$  with graphitic phases high in energy.



2498

2502

## A novel, selective free-radical carbamoylation of heteroaromatic bases by Ce(IV) oxidation of formamide, catalysed by *N*-hydroxyphthalimide

Francesco Minisci,\* Francesco Recupero, Carlo Punta, Cristian Gambarotti, Fabrizio Antonietti, Francesca Fontana and Gian Franco Pedulli



The Ce(IV)-NHPI system was used to generate a carbamoyl radical by oxidation of formamide; this nucleophilic radical has been successfully used in the carbamoylation of heteroaromatic bases.

# **3,4-Alkylenedioxy ring formation** *via* **double Mitsunobu reactions: an efficient route for the synthesis of 3,4-ethylenedioxythiophene (EDOT) and 3,4-propylenedioxythiophene (ProDOT) derivatives as monomers for electron-rich conducting polymers**

Kyukwan Zong, Luis Madrigal, L. "Bert" Groenendaal and John R. Reynolds\*





Mitsunobu chemistry has been employed as a new method for the synthesis of 3,4-alkylenedioxythiophenes as precursors to monomers for conjugated and electrically conducting polymers, including the commercially important 3,4-ethylenedioxythiophene (EDOT).

## Experimental and computational study of a reductive elimination mechanism in a methyl–Pd( $\Pi$ )–CNC carbene complex

David J. Nielsen, Alison M. Magill, Brian F. Yates, Kingsley J. Cavell,\* Brian W. Skelton and Allan H. White

Experimental and density functional studies on the decomposition of a palladium–methyl complex of the rigid CNC ligand 2,6-bis(1-alkylimidazolin-2-yliden-3-yl)pyridine show that reductive elimination to give 2-methylimidazolium species is a facile reaction.

## A new synthetic method to Mn carboxylate clusters: reductive fragmentation of $[Mn_{12}O_{12}(O_2CR)_{16}(H_2O)_4]$ to $[Mn_8O_2(O_2CR)_{14}(RCO_2H)_4]$ (R = CH\_2Bu')

Colette Boskovic, John C. Huffman and George Christou\*

Reduction of  $[Mn_{12}O_{12}(O_2CCH_2Bu')_{16}(H_2O)_4]$  followed by recrystallization from  $CH_2Cl_2/MeNO_2$  gives the lower-nuclearity product

 $[Mn_8O_2(O_2CCH_2Bu')_{14}(Bu'CH_2CO_2H)_4].$  This reductive fragmentation of a large, preformed cluster represents a promising new route to clusters not available by direct synthesis.

## $Photophysics \ of \ Ir(III) \ complexes \ with \ oligo(arylene \ ethynylene) \ ligands$

Ksenija D. Glusac, Shujun Jiang and Kirk S. Schanze\*

A photophysical investigation of two Ir(III) complexes that contain oligo(arylene ethynylene) ligands is reported.

600 700

500 600 700 Wavelength/nm 700

0.4



v





#### Synthesis of double-conjugated-segment molecules and their application as ultra-broad two-photon-absorption optical limiters

Junxiang Zhang, Yiping Cui,\* Mingliang Wang and Juzheng Liu

A series of novel double-conjugated-segment molecules, in which two conjugated segments are separated by an ether chain, were synthesized; these compounds provide a very broad two-photon absorption spectral range, which satisfies an urgent need in the optical limiting area.

## Silica gel catalysed generation of episulfonium ions: a mild method for the synthesis of heterocycles

Lorenzo Caggiano, David J. Fox and Stuart Warren\*



 $\beta$ -Carbonyloxy sulfides fragment when heated with silica or alumina to produce reactive episulfonium ion intermediates. Intramolecular activation of the leaving group and concurrent protection of a nitrogen or oxygen nucleophile via a cyclic carbamate or carbonate leads to the formation of pyrrolidines or cyclic ethers.

X = NH or O

2528

2530

2532

2534

## Lack of evidence of dilution history-dependence upon solute aggregation in water. A nuclear magnetic resonance determination of self-diffusion coefficients



Fernando Hallwass,\* M. Engelsberg and A. M. Simas

Unexpected dilution history-dependent aggregation effects, reported in Chem. Commun., 2001, 2224 for aqueous solutions, were found to be inconsistent with our nuclear magnetic resonance pulsed field gradient diffusion measurements.

## Dilithio-1,1,2-tris(diphenylphosphinoyl)ethane-1,2-diide: the first formal 1,2-dicarbanion stabilised by phosphorus?

Keith Izod,\* William McFarlane and William Clegg



The tris(phosphine oxide)  $\{Ph_2P(O)\}_2CHCH_2P(O)Ph_2$  readily undergoes deprotonation at adjacent carbon atoms to give the remarkable dimeric cluster

 $[(THF)Li({Ph_2P(O)}_2CCH{P(O)Ph_2})Li]_2.3PhMe (1), after$ recrystallization from toluene.



Varinder K. Aggarwal,\* Alessandra Lattanzi and Daniel Fuentes



Camphor-derived 1,3-oxathianes react with dichloroketene to give macrocyclic thiolactones with complete transfer of chirality.



Coupling dirhodium units through terpyridine bridges: synthesis and structure of a novel molecular rectangle

Jitendra K. Bera, Cristian Saul Campos-Fernández, Clérac Rodolphe and Kim R. Dunbar\*

The structure of a molecular rectangle  $[Rh_4(\mu-O_2CMe)_2(tppz)_2(MeOH)_4]^{4+}$  with edges defined by short Rh–Rh and long Rh–tppz–Rh units has been established.

## Formation and photophysics of a stable concave–convex supramolecular complex of $C_{60}$ and a substituted *s*-triazine derivative

David I. Schuster,\* Joel Rosenthal, Shaun MacMahon, Peter D. Jarowski, Christopher A. Alabi and Dirk M. Guldi\*

Spectroscopic, electrochemical and computational data show that  $C_{60}$  and a highly phenylated *s*-triazine derivative form a stable supramolecular complex at micromolar concentrations in solution at ambient temperatures, due to strong van der Waals attraction between their complementary surfaces.

## Hetero-metallomacrocyclic hosts that bind molecular guests in water

Paul de Wolf, Sarah L. Heath\* and Jim A. Thomas\*



.OMe

ÔMe

2540

2542

R

(R)-menthofuran

TiCL - Bu<sub>2</sub>N

The synthesis of metallomacrocycles 2 and 3 is reported. Photophysical studies indicate that energy transfer within the macrocylic structure is efficient. While preliminary host–guest studies reveal that both 2 and 3 are hosts for specific aromatic molecules.

# Efficient one-step synthesis of trialkylsubstituted 2(5H)-furanones utilizing direct Ti-crossed aldol condensation and its application to the straightforward synthesis of (*R*)-mintlactone and (*R*)-menthofuran

Yoo Tanabe,\* Kumi Mitarai, Takahiro Higashi, Tomonori Misaki and Yoshinori Nishii

Trialkylated 2(5*H*)-furanones, *e.g.* (*R*)-mintlactone and (*R*)-menthofuran, were prepared utilizing TiCl<sub>4</sub>–Bu<sub>3</sub>N mediated aldol condensation of ketones with  $\alpha$ , $\alpha$ -dimethoxyketones in a one-pot manner.

## A free energy minimisation study of the monoclinic–orthorhombic transition in MFI zeolite

Ricardo Grau-Crespo,\* Enrique Acuay and A. Rabdel Ruiz-Salvador

Free energy minimisation calculations of the crystal structure of the zeolite ZSM-5 are shown to reproduce the observed phase transition from monoclinic to orthorhombic symmetry.



(R)-mintlactone



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2556

2558

2560

## **PEG-Linked** luminescent platinum(II) complex as aqueous polymeric molecular light switch for protein binding reactions

Chi-Ming Che,\* Jun-Long Zhang and Li-Rong Lin

Attachment of poly(ethylene glycol) (PEG) to  $[Pt(4-HOPh-C^N^N)Cl]$ *via* covalent etheric bonds gives the luminescent polymer PEG-[Pt] (**2**), the photoluminescence of which is enhanced in hydrophobic regions of protein molecules (binding constant up to  $10^4 \text{ M}^{-1}$ ) in aqueous solution.







to the



Robert Haigh, K. M. Abdul Malik and Paul D. Newman\*

A new sterically encumbered pentacyclic phosphine derived form (1*R*)-camphor has been prepared and some aspects of its coordination chemistry with palladium(II) examined. The ligand has a rigid, multichiral skeleton with eight stereogenic centres. Upon reaction with  $Pd_3(OAc)_6$ , the ligand cyclometallates generating two further stereogenic centres (one carbon, one phosphorus) stereospecifically.

## [60]Fullerene-linked gold nanoparticles: synthesis and layer-by-layer growth on a solid surface

Young-Seok Shon\* and Hosun Choo

The facile synthesis of soluble and isolable [60]fullerene-linked gold nanoparticles and layer-by-layer assembly of  $C_{60}$ /nanoparticle films on the solid surface were studied.

## Structure–property trends in $\pi$ -stacked dithiazolo-dithiazolyl conductors

Leanne Beer, Jaclyn L. Brusso, A. Wallace Cordes, Erika Godde, Robert C. Haddon, Mikhail E. Itkis, Richard T. Oakley\* and Robert W. Reed

The resonance stabilized dithiazolo-dithiazolyl radical adopts a slipped  $\pi$ -stack structure exhibiting weak 1-D ferromagnetic coupling; variable temperature conductivity measurements indicate  $\sigma_{RT} = 2 \times 10^{-6} \text{ S cm}^{-1}$ .

## Synthesis of gallium phosphide nanowires via sublimation method

Hee Won Seo, Seung Yong Bae, Jeunghee Park, Hyunik Yang and Sangsig Kim

Gallium phosphide nanowires, with mean diameter of 40 nm and length up to  $300 \,\mu$ m, were synthesized by sublimation of ball-milled powders.





2568

2570

Quantitative formation and clean metal exchange processes of large void (>5000  ${\rm \AA}^3)$  nanobox structures

Michael Schmittel,\* Horst Ammon, Venkateshwarlu Kalsani, Andreas Wiegrefe and Christoph Michel

Using the HETPHEN concept a quantitative metallosupramolecular selfassembly process leading to large void nanoboxes was established. The unprecedented conversion of the  $Ag_4^{4+} \rightarrow Cu_4^{4+}$  nanobox convincingly underlines the reliability, robustness and flexibility of our approach.

## Wet chemical modification of PTFE implant surfaces with a specific cell adhesion molecule

Christoph Löhbach, Udo Bakowsky, Carsten Kneuer, Dieter Jahn, Thomas Graeter, Hans-Joachim Schäfers and Claus-Michael Lehr

The surface of PTFE based implant material could be covalently functionalised with adhesion molecules for improved bio-adhesion by wet chemical oxidation to introduce hydroxy groups followed by cross-linking with cyanuric chloride.

## High-valent metalloporphyrin, Fe(tpp)OTf, catalyzed rearrangement of $\alpha$ , $\beta$ -epoxy ketones into 1,2-diketones

Kohji Suda,\* Kenji Baba, Shin-ichiro Nakajima and Toshikatsu Takanami

Iron(III) tetraphenylporphyrin triflate, Fe(tpp)OTf, works as an efficient and characteristic Lewis acid catalyst in the selective rearrangement of  $\alpha$ , $\beta$ -epoxy ketones into 1,2-diketones.

#### Ring opening metathesis polymerisation in donor solvents

Christian Slugovc,\* Sandra Demel and Franz Stelzer

A screening concerning the functional group tolerance of the 'Super Grubbs' initiator (1) reveals that nitriles, amines or thiocyanates do not prevent ring opening metathesis polymerisation but strongly influence polymer properties.

Additive: MeCN, PhCN, HNEt<sub>2</sub>, NEt<sub>3</sub>, pyridine, etc.

Additive

Mes

ROOC

COOR



OTf

Fe(tpp)OTf

Meg

COOR

COOR

CI

CI.

Cy<sub>2</sub>F

#### A specific receptor of biological cystine polyion: distance-selective extraction and efficient chirality sensing with an ytterbium porphyrinate tweezer

Hiroshi Tsukube,\* Nobuyuki Tameshige, Satoshi Shinoda, Satomi Unno and Hitoshi Tamiaki

An ytterbium porphyrinate dimer acts as a new class of tweezer-type receptor, which offers selective extraction of biological cystine polyion and chirality sensing with circular dichroism spectroscopy.



## Electrochemical construction of an alternating multi-layered structure of palladium and gold nanoparticles attached with biferrocene moieties

Mami Yamada and Hiroshi Nishihara\*

A new convenient method for constructing novel hetero-layered films of different metal nanoparticles was developed, and a specific electrochemical behavior of hetero-layers of metal nanoparticles in acidic solution was found.

## Counter-ion effects switch ligand binding from C-2 to C-5 in kinetic carbenes formed from an imidazolium salt and $IrH_5(PPh_3)_2$

Anes Kovacevic, Stephan Gründemann, John R. Miecznikowski, Eric Clot,\* Odile Eisenstein\* and Robert H. Crabtree\*

Changing the counter-anion in 2-pyridylmethyl imidazolium salts (Br,  $BF_4$ ,  $PF_6$ ,  $SbF_6$ ) causes their kinetic reaction products with  $IrH_5(PPh_3)_2$  to be switched from normal C-2 to abnormal C-5 binding.

## Nonionic oligomeric polymer directed synthesis of highly ordered large pore periodic mesoporous organosilica

Abdelhamid Sayari\* and Yong Yang

Ethane-bridged periodic mesoporous silica with large pore (5.5 nm), high surface area, and highly ordered 2D hexagonal structure has been synthesized reproducibly in high yield using nonionic oligomeric Brij 76 polymer as structure-directing agent and 1,2-bis(triethoxysilyl)ethane (BTEE) as organosilica source in acidic media at 50  $^{\circ}$ C.

## 2584

10.00 nm

depends on anion

2582

## Simulation of TaqMan by two single-labelled probes

De-Ming Kong, Long Gu, Han-Xi Shen\* and Huai-Feng Mi

A novel method for duplex probes is designed to simulate the TaqMan probe during polymerase chain reaction. In this method, two single-labelled probes are used.



Electro-oxidation

of biferrocene units

in electrolyte - CH2Cl2

ITO electrode

Composite Pd/Au nanoparticle film

2580

Template

## Biocatalytic synthesis of uridine 5'-diphosphate *N*-acetylglucosamine by multiple enzymes co-immobilized on agarose beads

Jun Shao, Jianbo Zhang, Jozef Nahálka and Peng George Wang\*



Recombinant *N*-acetylglucosamine kinase, pyruvate kinase, *N*acetylglucosamine phosphate mutase, uridine 5'-diphosphate *N*acetylglucosamine pyrophosphorylase, and inorganic pyrophosphatase were overexpressed in *E. coli* and co-immobilized on agarose beads for the practical synthesis of uridine 5'-diphosphate *N*-acetylglucosamine.

## Microwave-assisted synthesis of carbon supported Pt nanoparticles for fuel cell applications

Wei Xiang Chen, Jim Yang Lee\* and Zhaolin Liu

Carbon supported Pt nanoparticles were prepared by microwave irradiation. TEM images demonstrate that the Pt nanoparticles were uniform in size and shape. Electrochemical experiments showed that the Pt/C catalyst exhibited very high electrocatalytic activity for oxidation of liquid methanol.

#### Isotopic changes during the synthesis of amphetamines

James F. Carter,\* Emma L. Titterton, Helen Grant and Richard Sleeman



2590

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There is currently a great deal of interest in the use of isotopic characterisation of drugs of abuse as a means to identify their source and synthetic pathway. A comparison of amphetamines, prepared *via* reductive amination, and their precursors reveals kinetic isotope effects which result in small variations in  $\delta^{13}$ C and large variations in  $\delta^{15}$ N. The product amphetamines, therefore, reflect the reaction conditions rather than the starting materials.

#### ADDITIONS AND CORRECTIONS

Hiroshi Katagiri, Nobuhiko Iki, Yoshiaki Matsunaga, Chizuko Kabuto and Sotaro Miyano 'Thiacalix[4] aniline' as a highly specific extractant for  ${\rm Au}({\rm III})$  and  ${\rm Pd}({\rm II})$  ions

## CONFERENCES

Dates, venues and contact details of forthcoming events.

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Highlights from the 37th ESF/EUCHEM Conference on Stereochemistry, Bürgenstock, Switzerland, April 2002

Patrick Guiry and Florian Hollfelder

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